25p

X

(NASA   TMX-5-4537)

A PRELIMINARY STUDY OF THE SOLID-STATE BONDING

OF THORIA DISPERSION STRENGTHENED NICKEL

T. T. Bales and C. R. Manning, Jr.   [1964] 25p

NASA Langley Research Center,
Langley Station, Hampton, Va.

Presented at the ~~Eighth~~ 8'th Meeting of the
Refractory Composites Working Group

Fort Worth, Texas
January 14-16, 1964

A PRELIMINARY STUDY OF THE SOLID-STATE BONDING

OF THORIA DISPERSION STRENGTHENED NICKEL

By T. T. Bales[*] and C. R. Manning, Jr.[*]

NASA Langley Research Center

SUMMARY

A preliminary investigation was made to determine the feasibility of

joining a nickel-thoria composite by solid-state or diffusion bonding. The

parameters for bonding in the solid state for thoriated nickel were estab-

lished at temperatures of 2,000° F and 2,150° F. Bonding parameters were

similarly established for the joining of thoriated nickel to a precipitation

strengthened superalloy at 1,850° F. The basic specimen for determining the

efficiency of the bond consisted of an overlapped tensile-shear specimen. Two

structural panels and a high-temperature engine blade were also bonded to

demonstrate the practical aspects of this joining method. A metallographic

study of the bonded joints as well as a description of the equipment and

bonding procedures utilized in the investigation are included.

INTRODUCTION

A continual requirement exists for new or improved metals and metal-ceramic

composites that will withstand the severe environments associated with advance

propulsion and thermal protection systems of aerospace vehicles. The materials

required for such applications must possess adequate elevated-temperature

strength, display good ductility, retain resistance to oxidation, and be capa-

ble of being formed and joined into useful hardware. One material that has

---

[*]Aerospace Technologist

shown promise in this respect for temperatures ranging from 1,700° F to 2,300° F is a nickel-thoria composite shown in figure 1. This material is known as thoria dispersion strengthened nickel. The material contains a nickel matrix and an inert second phase of thoria indicated by the small particles. Before the full potential of this material or any similar type of material can be utilized for structural application it is imperative that a suitable method of joining be available. Unfortunately, composite materials such as the thoriated nickel do not readily lend themselves to joining by conventional welding methods. Difficulties are encountered in joining this type of composite materials because the inert second phase agglomerates in the weld area. This result is shown in figure 2. The photomicrograph of thoriated nickel joined by electron beam welding shows an agglomeration of the thoria particles in the weld area. This agglomeration of the thoria produced a weld of relatively low strength. (See ref. 1.) This problem of agglomeration can be avoided by joining the material in a solid rather than molten state. Figure 3, for example, shows a joint produced in thoriated nickel by solid-state or diffusion bonding. It is apparent that no thoria agglomeration occurred and that a good metallurgical bond was obtained. Diffusion bonding thus appears to offer a possible method for joining thoriated nickel.

Because diffusion bonding appears to possess certain advantages in joining composite materials over more conventional approaches, the National Aeronautics and Space Administration at the Langley Research Center is currently conducting a preliminary investigation of solid-state bonding techniques. The study included joining of simple overlapped tensile-shear specimens and also included more complex specimens such as structural sandwich panels. The specimens are being subjected to various tests to determine strength and joint integrity. In

2

addition, a metallurgical study of the joining process is being made and some indication of the joining parameters, namely, temperature, time, and pressure, are being obtained.

MATERIALS AND SPECIMENS

The sheet materials included in this investigation to date have included a composite of 2-percent thoria and 98-percent nickel by weight and a precipitation strengthened nickel-base superalloy. The nominal thickness of each material selected for the study is 0.020 inch.

Several types of specimens are currently being utilized in this investigation. The first type designed to give basic data on the diffusion bonding process is a single overlap specimen shown in figure 4. This specimen is used to obtain the shear strength of the bonded joint. The metal strips are cleaned with acetone and water before bonding. The desired overlap is maintained in the specimen by making a tack weld at the corners of the overlap by means of a capacitor discharge machine. The second specimen shown in figure 4 is a shear specimen used to determine the basic shear strength of the metal.

The tensile-shear specimens were designed so that failure by shear of the joint would occur. An overlap distance equal to twice the sheet thickness was found to be satisfactory. Eccentricity of loading in this type of simple joint is undesirable and unavoidable. No attempt was made to adjust or compensate the test results on the strip specimen for the eccentricity of loading.

Another type of specimen fabricated in this study of solid-state bonding included several small structural panels. The panel shown in the upper part of figure 5 was fabricated from 0.025-inch-thick thoriated-nickel sheet while the

multilayer panel in the lower portion of the figure was made from 0.0025-inch-thick dimpled sheet, 0.004-inch-thick interior face sheets, and 0.010-inch-thick external face sheets. The specimen shown in figure 6 is a high-temperature engine blade. This component was fabricated from 0.050-inch-thick thoriated-nickel sheet. The joint produced by diffusion bonding is indicated.

BONDING PROCEDURES

Two different procedures were utilized in the solid-state bonding of these materials. One procedure involved the application of pressure and heat for various times and required the use of reduced pressure (vacuum) to prevent oxidation of the faying surfaces. In the second procedure the specimen was subjected to pressure, heat was provided by the direct resistance heating of the part to be joined, and the assembly was surrounded by an inert gas, argon, rather than vacuum.

The fixture or bonding device that was used to join the strip specimens is shown in figure 7. The pressure was applied by means of a 1/2-inch-diameter tungsten bolt tightened to a prescribed torque. The compressive load applied for any torque loading on the specimen was obtained from calibration runs at room temperature using a load cell. The fixture containing the specimens was then placed in the vacuum furnace and held at temperature for the necessary time. The fixture used for bonding the sandwich panels is shown in figure 8. This assembly consists of a stainless-steel retort. Pressure on the specimen was obtained by clamping the specimen against the base of the retort. The retort was welded closed and connected to a pumping system to prevent oxidation of the metal. The assembly was then placed in a furnace for bonding.

Another approach for bonding utilized in this study is shown in figure 9. This setup utilized resistance heating of the specimen and argon gas to prevent oxidation. The short strips were placed in the fixture with each end attached to a water-cooled power lead. Compressive load was applied by means of a ceramic ram using a 10,000-pound screw powered testing machine. The sheet strips were then heated to the bonding temperature in approximately 2 minutes by resistance heating. Argon gas was flowed through the quartz tube during the bonding operation. The bonding temperatures were maintained in this setup for times ranging from 1 to 10 minutes.

RESULTS AND DISCUSSION

Bonding Parameters

Initial approaches to the diffusion bonding of thoriated nickel (ref. 1) indicated that the material could be bonded successfully in a furnace using the welded stainless-steel retort described in the preceding section. The pressure utilized in the retort was 1×10^{-4} mm of Hg, the temperature was $2,000^{\circ}$ F, and the time 24 hours. Both of the sandwich panels shown in figure 5 and the jet engine blade shown in figure 6 were bonded by this technique. Further work on this material has been continued using a vacuum furnace with pressure ranging from 1×10^{-5} to 1×10^{-6} mm of Hg, temperature ranging from $2,000^{\circ}$ F to $2,100^{\circ}$ F and bonding time from 16 to 24 hours. Bonding pressures of 10,000 psi were applied. During the bonding study at $2,100^{\circ}$ F and above, a white residue formed on the surface of the thoriated nickel when the pressure was below 1×10^{-3} mm of Hg. This residue was considered to be thoria and hindered the solid-state bonding process.

In an effort to reduce the bonding times, solid-state resistance bonding techniques as previously described were attempted. The bonding parameters are indicated in figure 10 in which the shear strength of the bond is plotted against bonding pressure. Pressure in all cases shown here was maintained for 1 minute at temperatures of 2,000° F and 2,150° F. These results indicate a linear relationship between bonding pressure and strength of the joint up to a stress of approximately 35 ksi. After reaching a maximum shear stress of 35 ksi, which is approximately 70 percent of the base metal strength, the mode of failure changes to a tensile failure of the sheet. Because of eccentricity of loading and other effects, which have not been established, the failure of the sheet occurred near the bonded joint at a strength equal to 80 percent of the base metal tensile strength. Further efforts to explore the bonded joints at higher shear strengths would necessitate a smaller overlap on the basic tensile-shear specimen. Some further effort in this direction is contemplated. Note that bonding pressures of 12,500 psi and 8,000 psi at temperatures of 2,000° F and 2,150° F, respectively, produced joints that could withstand a shear stress of at least 35 ksi. As the bond pressure decreased the shear strength of the bonded joint also decreased. It was also noted that no residue formed on the surface of the specimens bonded above 2,100° F as had been noted for the specimens bonded in a vacuum. Two test points indicated by the dark symbols were obtained from specimens joined with the use of the tungsten fixture without resorting to resistance heating. These two results are compatible with the other data; however, note that the bonding time was 16 hours in contrast to the 1 minute utilized with the specimen heated by the resistance method.

Figure 11 shows the bonding parameters utilized for joining thoriated nickel to a precipitation strengthened nickel alloy at 1,850° F. The results indicate that the shear strength of the joint is a function of both pressure and bonding time. A similar result was not obtained for the thoriated nickel alone. Extrapolation of these linear results to the estimated strength of the thoriated nickel suggests that a good metallurgical bond between the thoriated nickel and the nickel-base superalloy can be obtained at bond pressures of 25,000, 17,000, and 13,000 psi in 1, 5, and 10 minutes, respectively. Figure 12 shows the microstructure of the thoriated-nickel diffusion bonded at 1,850° F to the precipitation strengthened superalloy. A good metallurgical bond appears to exist between the two materials. Figure 13 depicts a means of joining the materials at a lower temperature of 1,700° F by means of using a beryllium-copper interleaf. The compounds formed in each bond are being investigated for strength and ductility at the present time.

JOINT STRENGTH AND METALLURGICAL STUDY

The shear strength of the solid-state bonded materials was determined from tensile-shear tests at various temperatures to 2,000° F. These results for thoriated nickel are shown in figure 14 by the symbols. A solid line has been drawn through these data. Shown also are the ultimate tensile strength of the thoriated nickel indicated by the upper dotted curve and the estimated shear strength of the material indicated by the lower dotted curve. It is assumed that the ultimate shear stress is 60 percent of the ultimate tensile stress. This assumption was verified at room temperature where the shear strength of the metal was determined from the specimen shown in the lower half of figure 4. This test result is shown by the solid symbol. As noted previously, the strength

7

of the joints at room temperature was approximately 70 percent of the estimated shear strength of the material. This decrease in strength is probably attributed to the bending in the overlap joint during testing. A further decrease in strength is apparent at the higher temperatures, where at 1,200° F and 2,000° F for example, the bonded joint strength is only 60 percent and 40 percent, respectively, of the base material shear strength. This may be attributed to a combination of various phenomena: The tensile-shear strength of the material at elevated temperatures may not be equal to 60 percent of tensile strength found at room temperature. A thin nickel layer with a low thoria concentration may have formed at the original interface. This relatively pure nickel layer should exhibit a lower shear strength than the dispersion strengthened material at elevated temperatures, but would be expected to have approximately the same strength at room temperature. In order to verify the presence of the thin nickel layer and to establish the concentration of thoria in the area of the joint, further detailed metallurgical studies are required. An electron probe microanalyzer will be used in this additional study.

Figure 15 shows the microstructure of the bonded joint, after elevated-temperature testing. This figure shows a portion of the bonded area of one tensile strip after elevated-temperature shear test. It appears that the material delaminated beneath the original interface at the overlapped joint.

The role of surface finish or roughness has not been investigated to date. It may assume importance for certain types of materials. Some study of this item is contemplated. Further efforts will also be made to investigate the suitability of various interleaf materials and on the possibility of bonding metals, particularly nickel, that may have an oxidation-resistant coating on the surface.

CONCLUDING REMARKS

A study of several solid-state bonding techniques for the joining of thoriated nickel to itself and other nickel-base materials has been undertaken. The following conclusions are made from this preliminary investigation:

1. Solid-state joining techniques appear to be feasible for joining nickel dispersion strengthened materials.

2. A linear relationship exists between the bonding pressures and bond strength for thoriated nickel and a precipitation strengthened nickel superalloy.

3. Thoriated nickel can be bonded by solid-state diffusion in an argon atmosphere.

4. The amount of pressure necessary to form a satisfactory bond is time dependent for the thoriated nickel to nickel superalloy joints.

5. No thoria agglomeration occurred during the bonding process.

6. The time necessary to obtain a satisfactory bond apparently is greatly reduced if resistance heating is utilized.

7. A more detailed study should be made of the elevated temperature properties of diffusion bonded joints of composite materials.

REFERENCE

1. Manning, Charles R., Jr., Royster, Dick M., and Braski, David N.: An Investigation of a New Nickel Alloy Strengthened by Dispersed Thoria. NASA TN D-1944, 1963.
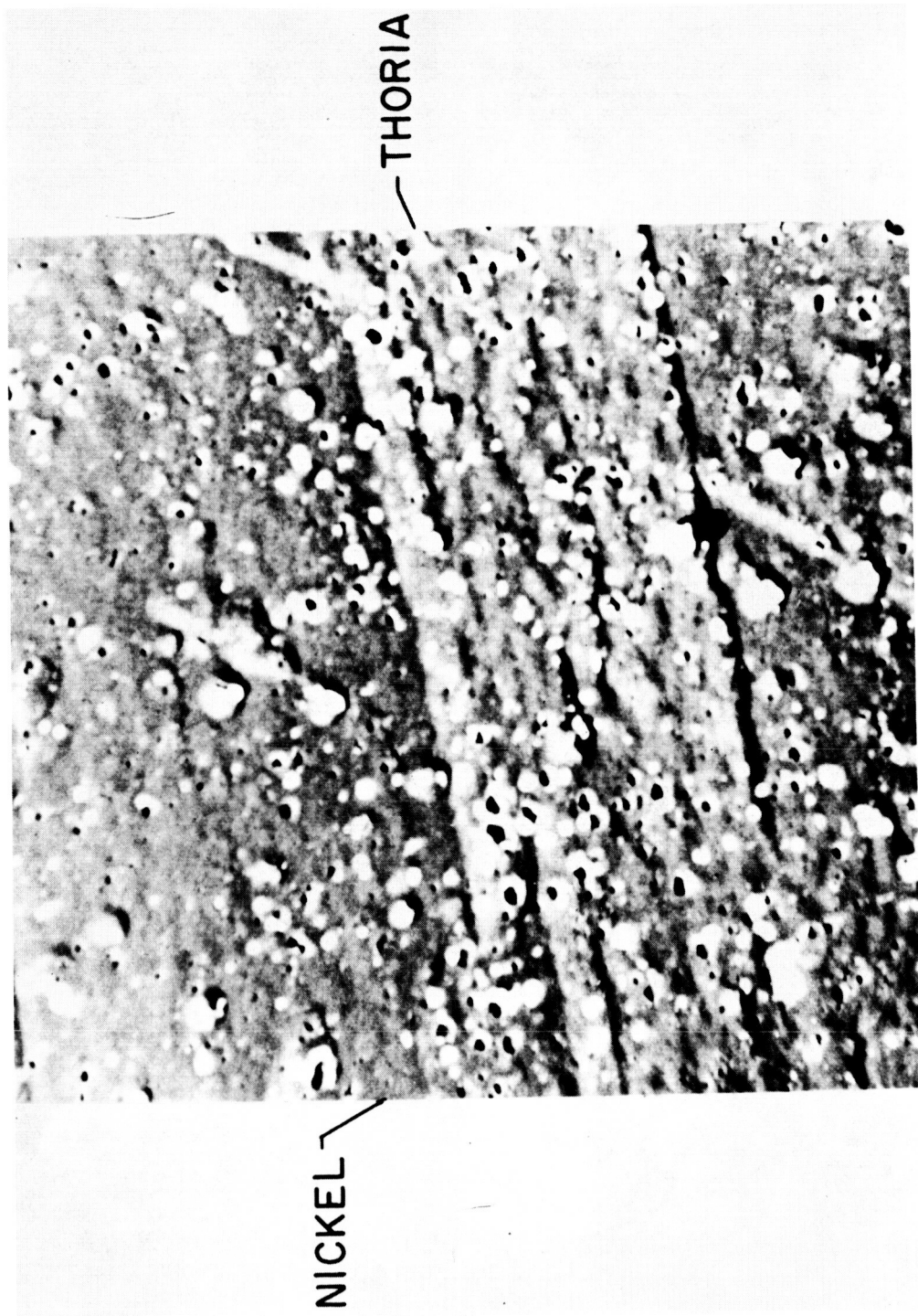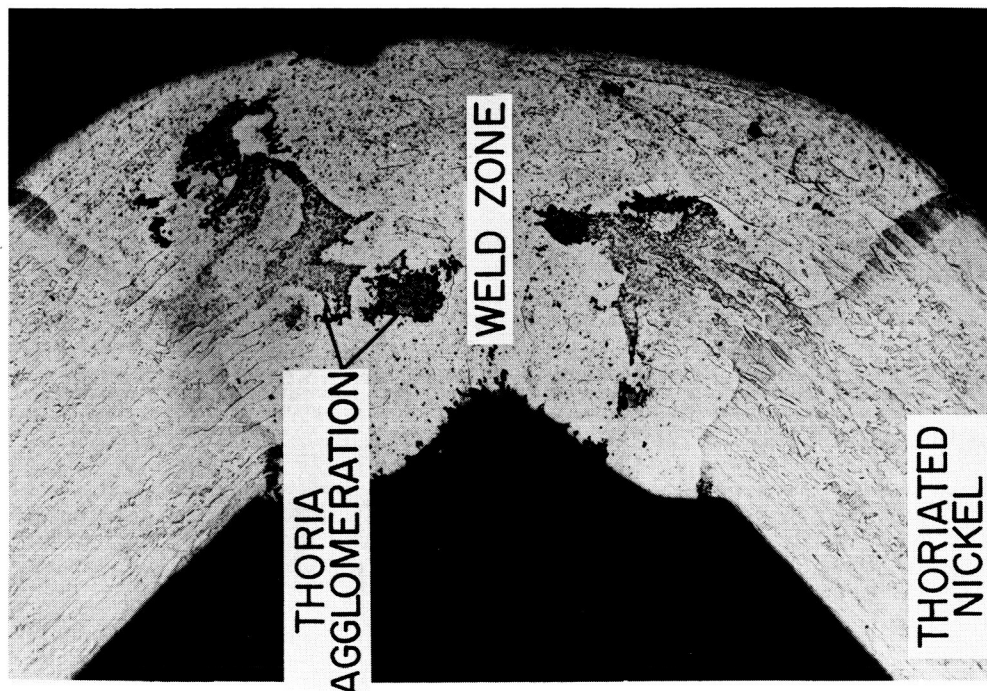
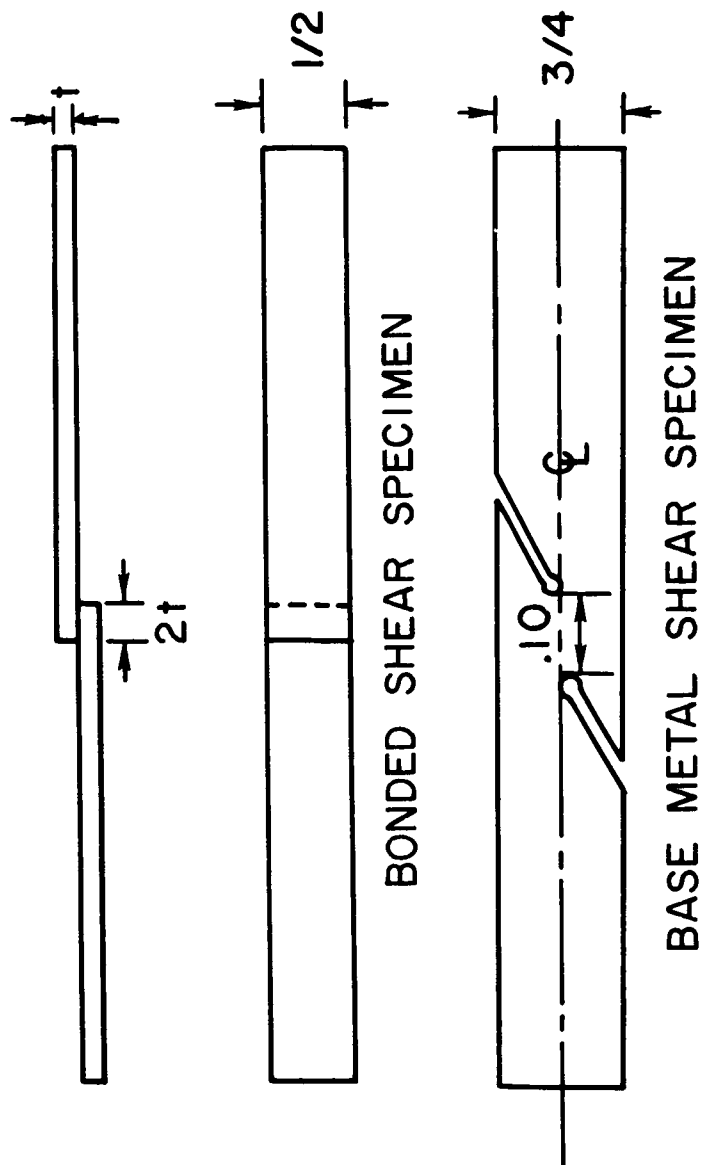Figure 1.- Electron micrograph of thoriated nickel, 10,000×.

THORIA AGGLOMERATION

WELD ZONE

THORIATED NICKEL

NASA

Figure 2.- A photomicrograph of thoriated nickel welded by the electron beam process, 150X.

Figure 3.- Diffusion bond in thoriated-nickel sheet, 500X.

t

$2t$

BONDED SHEAR SPECIMEN

1/2

.10

℄

3/4

BASE METAL SHEAR SPECIMEN

NASA

Figure 4.- Tensile shear specimens. All dimensions in inches.

(a) SINGLE-LAYER
PANEL

(b) MULTILAYER PANEL
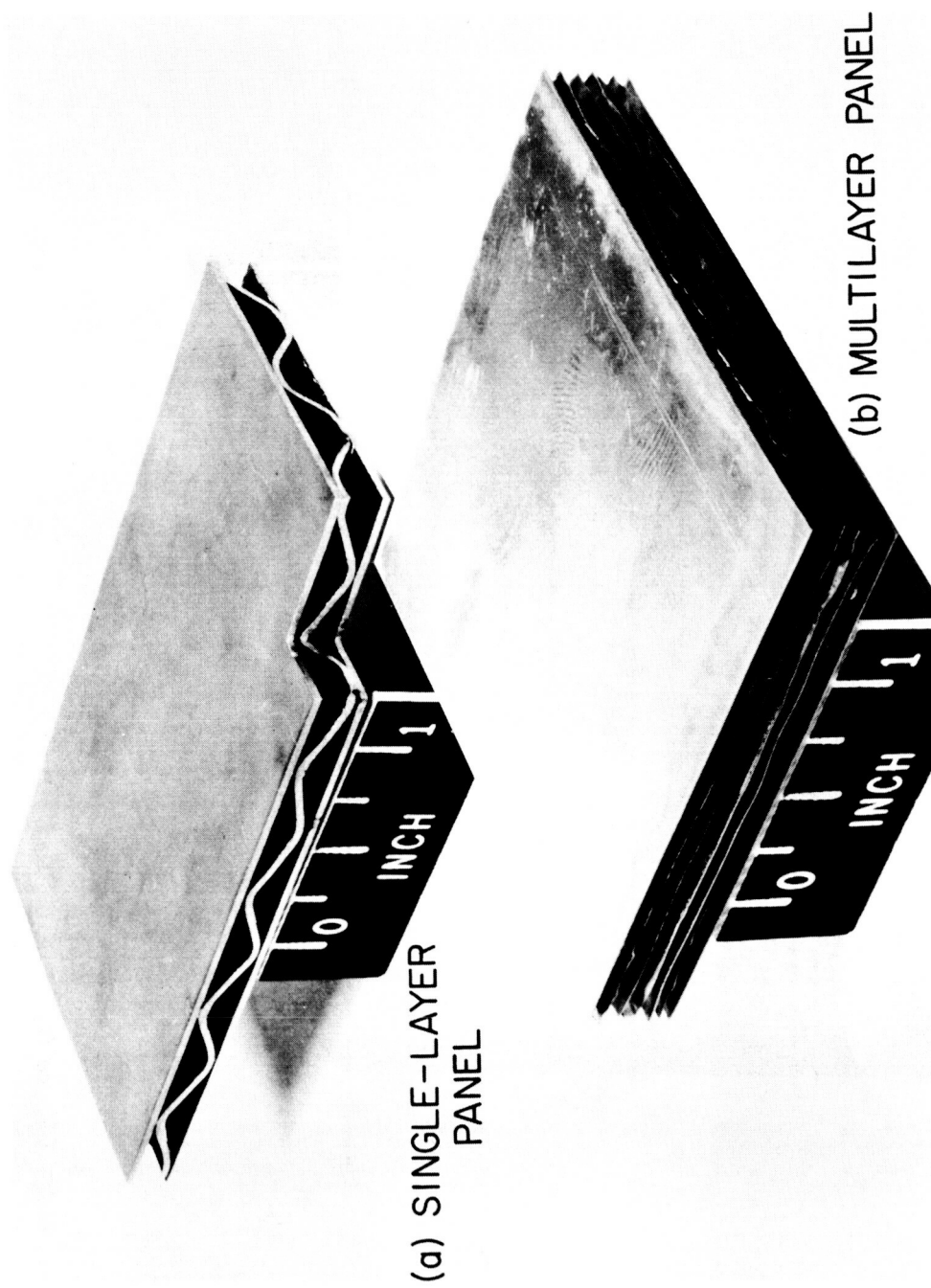
NASA

Figure 5.- Diffusion bonded thoriated-nickel structural panels.

BONDED JOINT

INCH
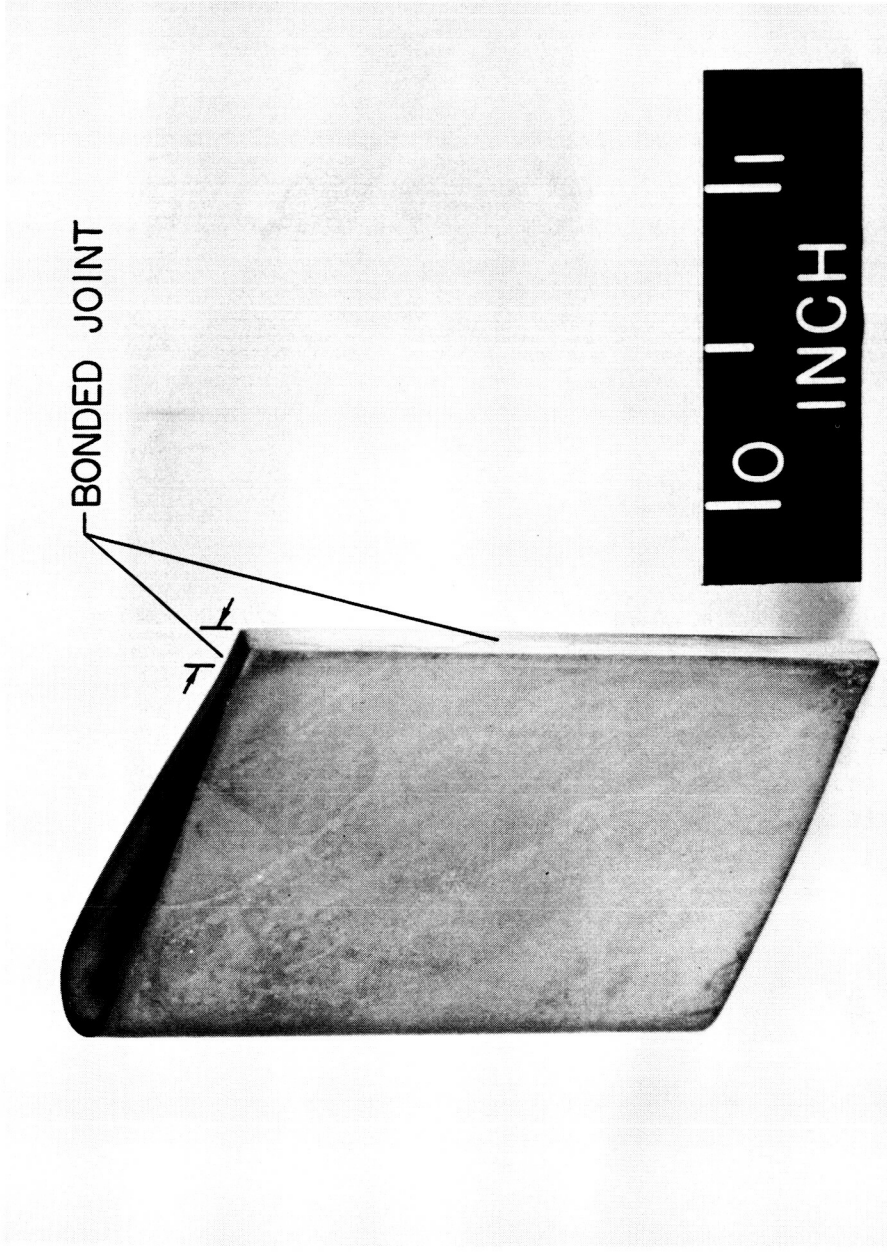
NASA

Figure 6.- Diffusion bonded thoriated-nickel jet engine blade.

TUNGSTEN BONDING CAPSULE

TUNGSTEN SCREW
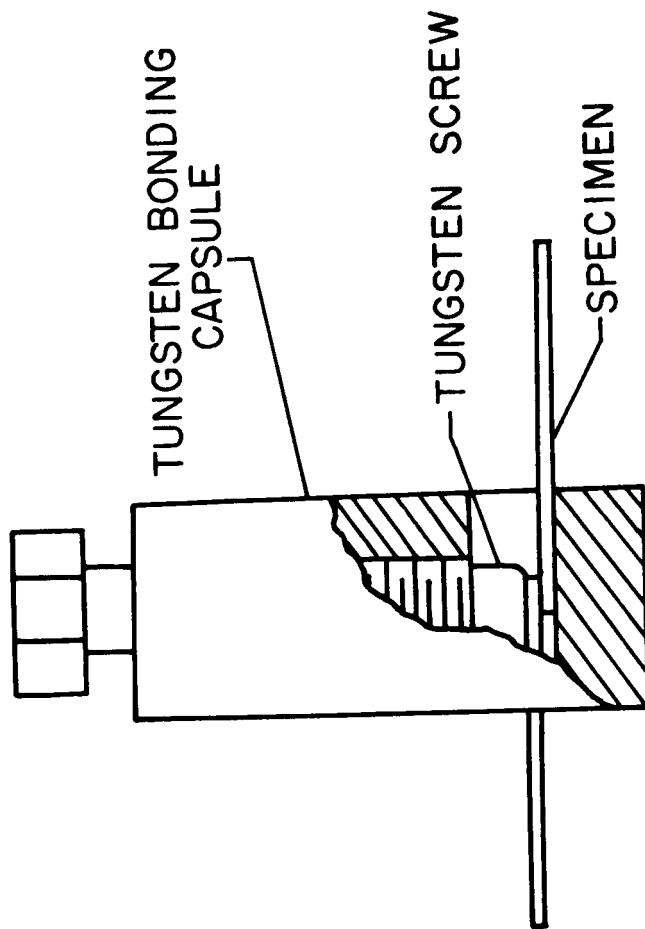
SPECIMEN

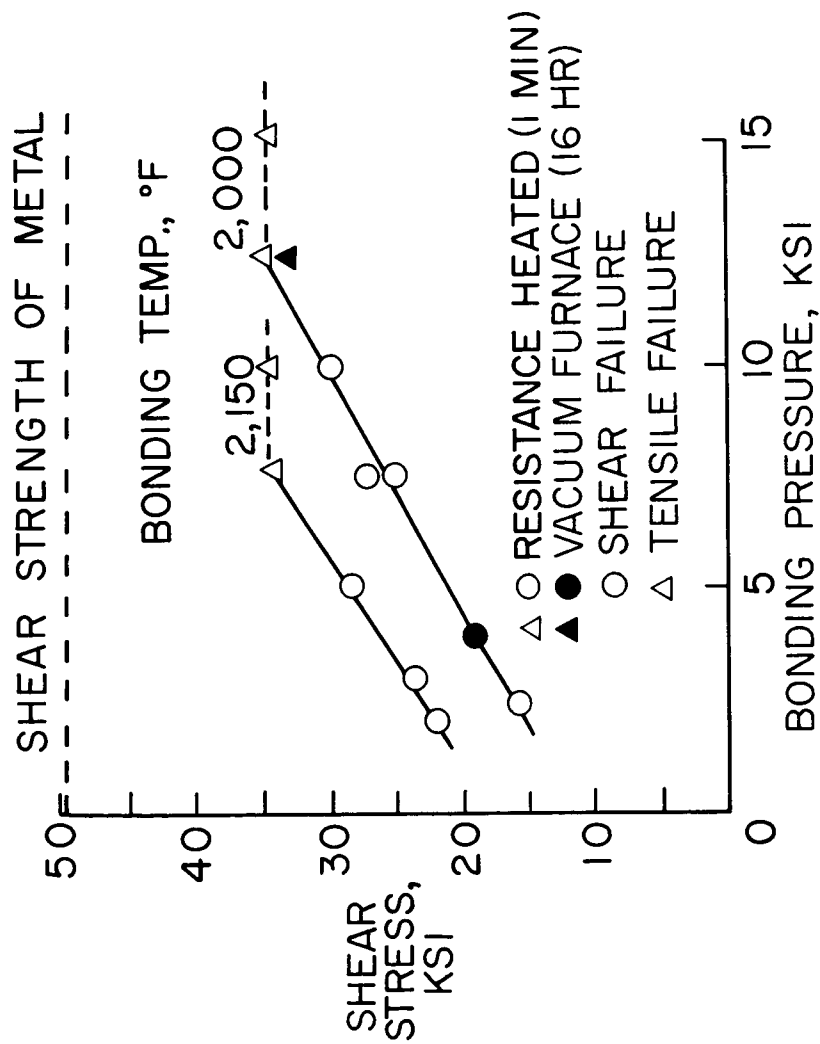NASA

Figure 7.- Bonding device for tensile shear specimen.
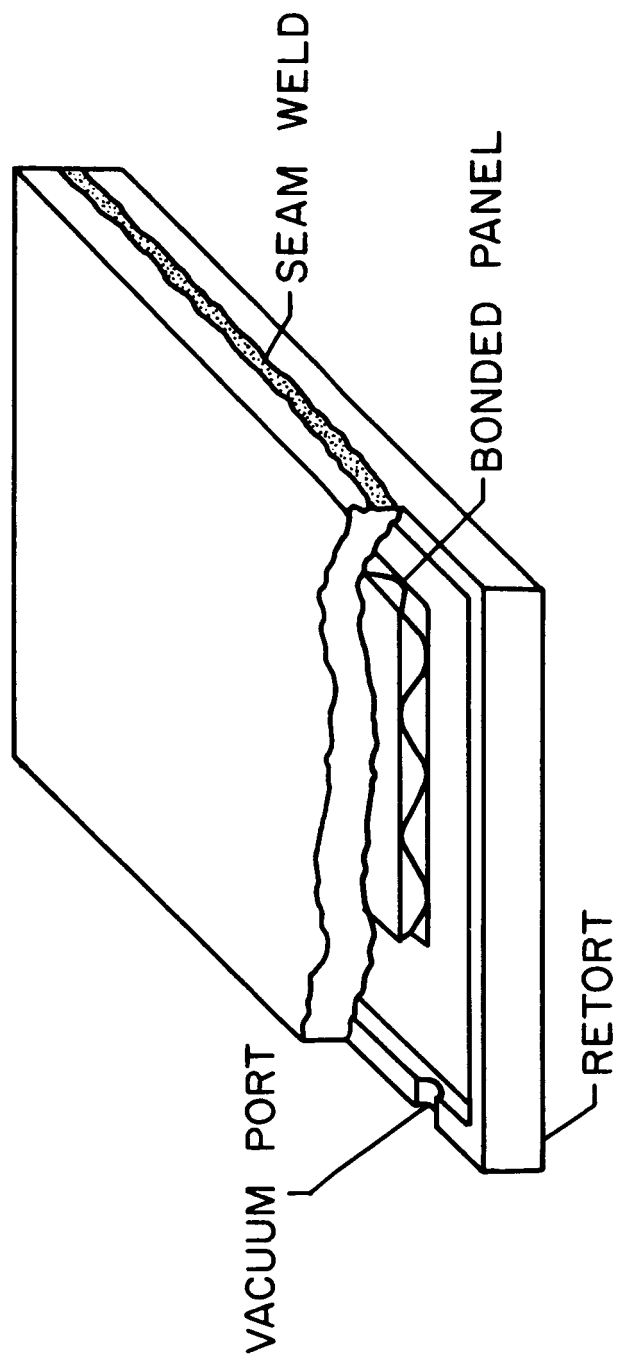
Figure 8.- Retort for diffusion bonding of structural panels and engine vanes.
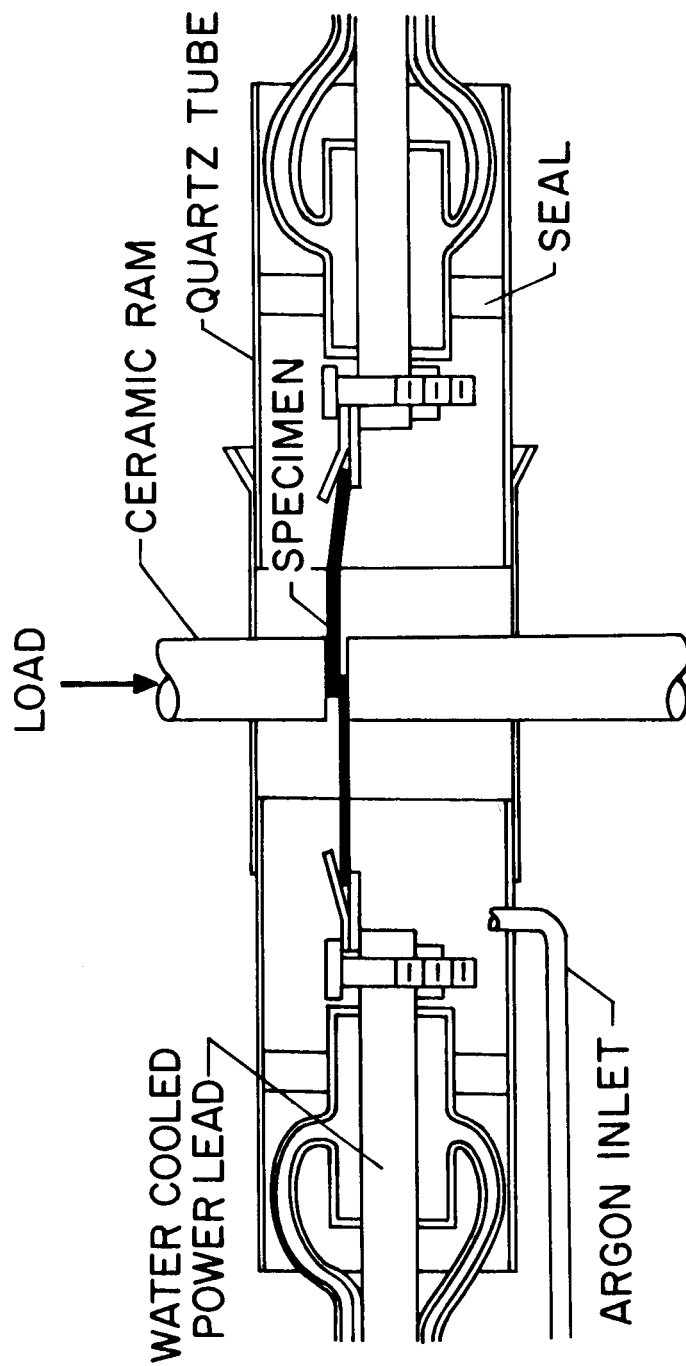
NASA

Figure 9.- Resistance heating apparatus for diffusion bonding of tensile shear specimens.

Figure 10.- Shear strength of bonded thoriated-nickel strips tested at room temperature.
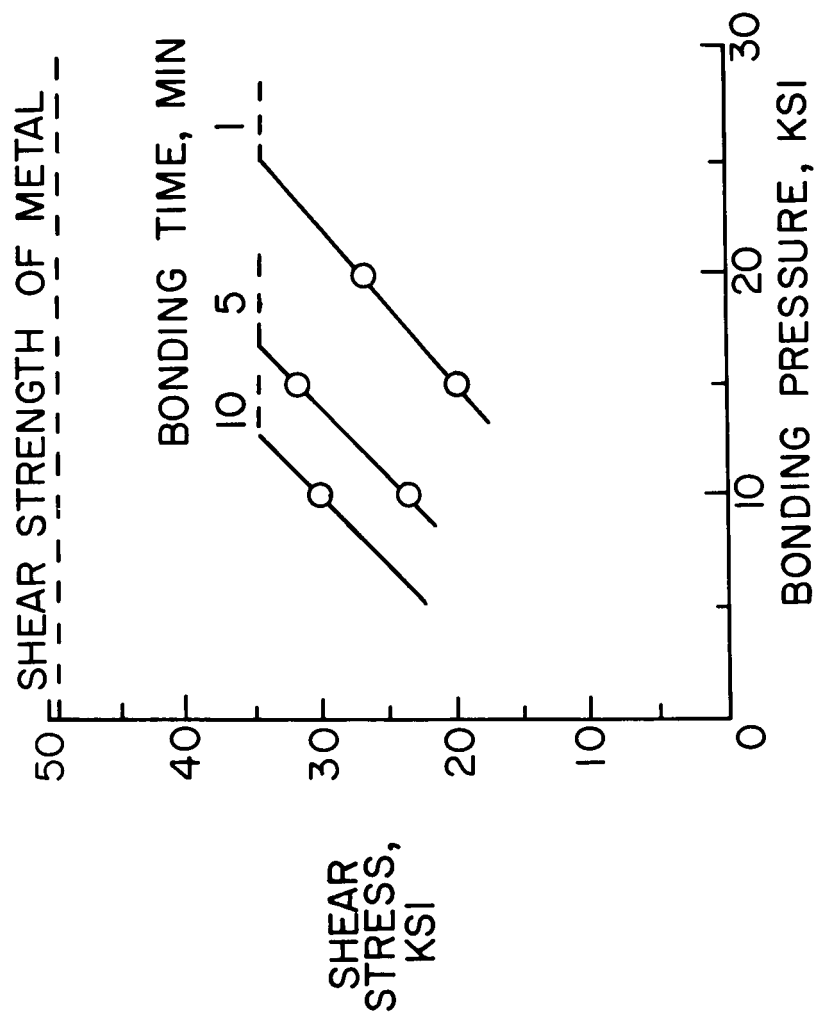
NASA

Figure 11.- Shear strength of bonded joints between thoriated nickel and a nickel base
superalloy bonding temperature 1,850° F.

NICKEL BASE SUPERALLOY

DIFFUSION BONDED
JOINT

THORIATED-NICKEL

NASA

Figure 12.- A photomicrograph of thoriated nickel bonded to a nickel base superalloy, 500×.

NICKEL BASE SUPERALLOY

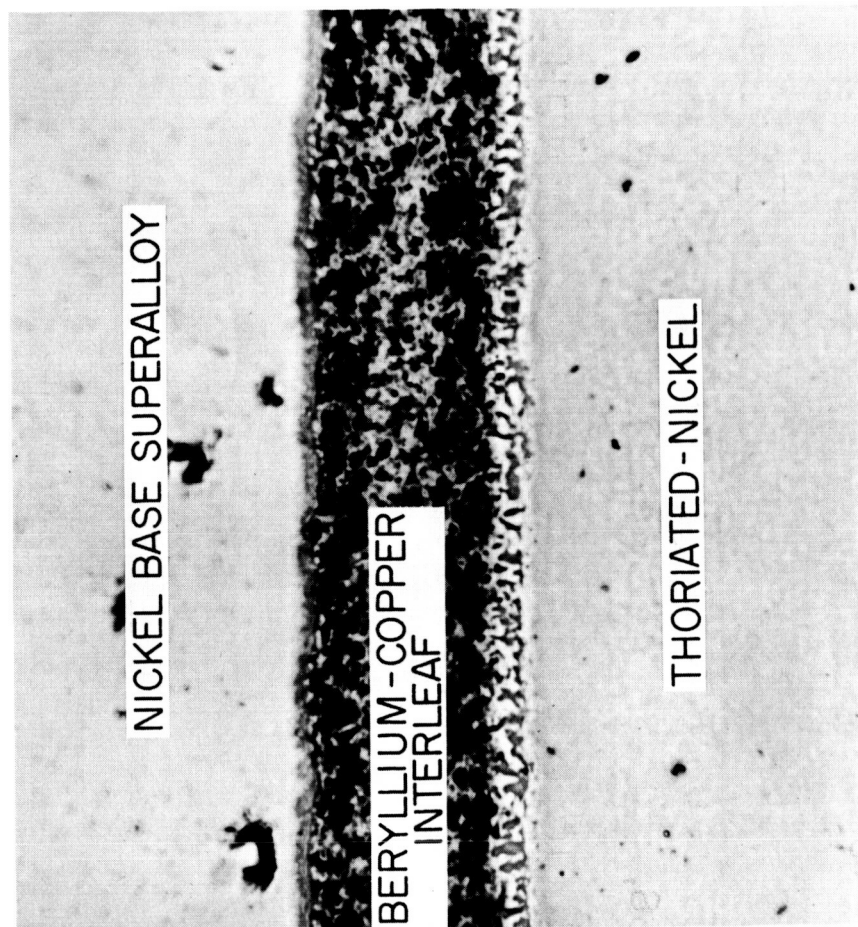BERYLLIUM-COPPER INTERLEAF

THORIATED-NICKEL

Figure 13.- A photomicrograph of thoriated nickel joined to a nickel base superalloy using a beryllium copper interleaf, 500X.
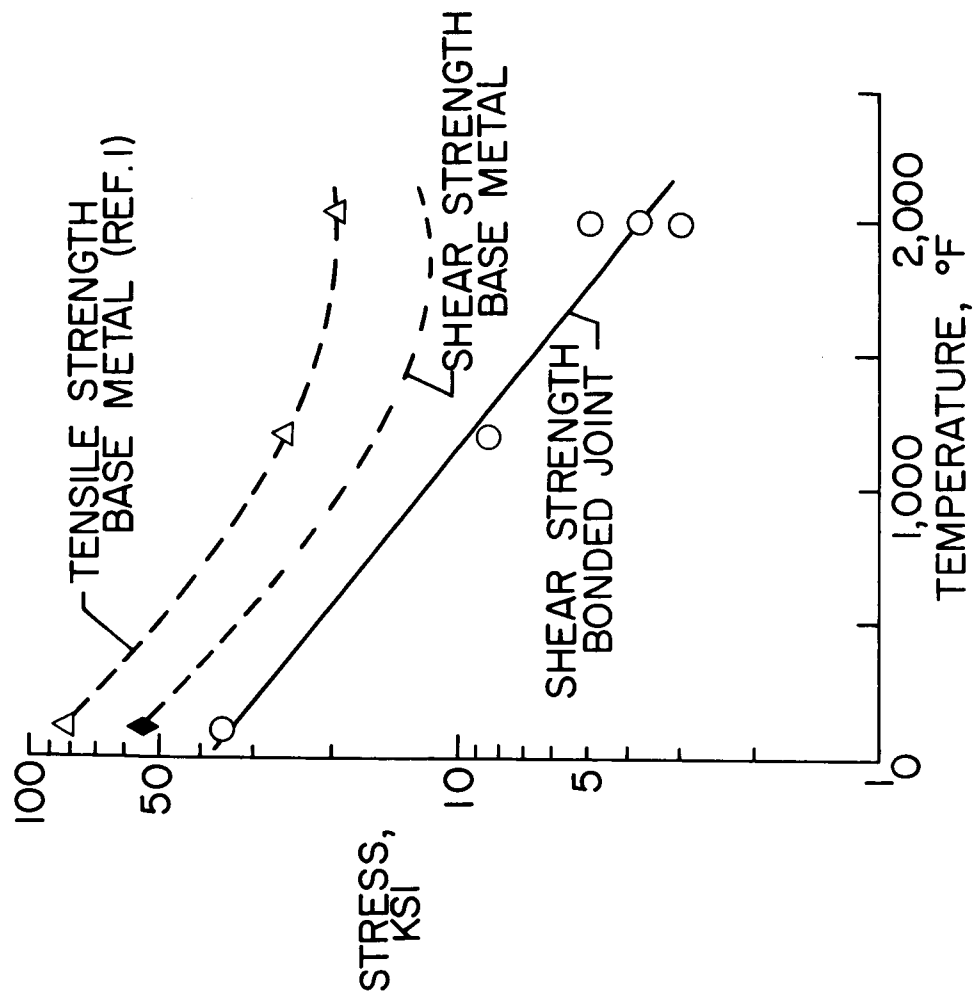
Figure 14.- Comparison between shear strength of thoriated-nickel joints at elevated temperatures and base metal.

NASA

Figure 15.- Microstructure of a bonded thoriated-nickel joint after elevated temperature shear failure, 375X.